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A Day in the Life of the Corrosion Laboratory of Cepel

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Abstract

A day in the life of the corrosion laboratory of Cepel is a dynamic experience with interdisciplinary activities involving several pieces of equipment, materials and analytical techniques aimed at understanding and preventing the effects of corrosion in assets of the Brazilian electric sector. This article presents highlighted activated of the day to life in the laboratory including surface and sample preparation, organic coatings, electrochemical techniques, accelerated corrosion tests, corrosivity of atmosphere and determination of corrosion rate, soil corrosion and quality control of galvanized steel.

Keywords

Corrosion, Electrochemistry, Coatings, Galvanized Steel

1. Introduction

Corrosion is a natural process that causes significant damage to metallic structures and equipment in the electric sector, which can lead to premature failure in service [1]. Therefore, the study of corrosion is essential in understanding how to prevent and mitigate its effects. It requires interdisciplinary expertise in materials science, chemistry and electrochemistry and a strong connection with other laboratories for cooperation purposes to solve corrosion problems. Corrosion laboratories play a crucial role in this field by testing the effectiveness of corrosion protection methods and by assisting in the development of highly corrosion-resistant materials.

The Electric Energy Research Center - Cepel[1] goal is to foster scientific and applied research and develop resources to improve the operation and reliability of the Brazilian power systems. Its portfolio of technological services and products boosts the power grid safety and effectiveness, contributing at all levels to the Brazilian society and industry. To overcome the challenge of developing solutions for such a large and complex electric sector, the Center features several laboratories and essential knowledge areas, including high voltage, high power, energy efficiency, chemistry, metallography, mechatronics, mechanics, and corrosion. In particular, the main research lines of Cepel's corrosion laboratory are electrochemistry, organic coatings, galvanized steel, atmospheric corrosion and materials selection.

The day-to-day life of the corrosion laboratory of Cepel is a dynamic experience full of activities that involve various types of equipment, materials and analytical techniques aiming to control the corrosion of metals. The laboratory relies on a team of corrosion experts, engineers and technicians who conduct experimental research, perform corrosion testing and provide consulting services. In summary, a usual day in the life of the laboratory involves sample preparation, setting up equipment and carrying out measurements and critical analysis of test-relevant quantities. Materials are exposed to corrosive environments, followed by the analysis of changes in their properties due to corrosion processes. Corrosion testing is often coupled with other techniques that are sensitive to these changes, including electrochemical tests, microscopic and chemical analysis. Some of these are carried out by other areas, such as metallography and chemistry laboratories, providing support to the corrosion laboratory to come up with solutions for corrosion prevention.

Many activities that are common for every laboratory are also accomplished, such as measuring systems or instruments calibration, sampling and labeling, results analysis and issuing reports. This paper focuses on the activities that make a corrosion laboratory unique. It is an invitation to know more about the day-to-day in the corrosion laboratory of Cepel and to dive into the world of corrosion processes.

2. Cepel's Corrosion Laboratory

The corrosion laboratory of Cepel has expertise in experimental research for understanding and preventing the effects of corrosion in metallic parts of assets of the Brazilian electric sector. It performs the following main activities:

- ✓ Corrosion testing in aggressive environments, such as salt spray, saline solution, soil and acid rain.
- ✓ Corrosion rate determination by mass loss and classification of the atmospheric degree of corrosivity.
- ✓ Electrochemical tests, namely potential monitoring, polarization and impedance spectroscopy.
- ✓ Surface preparation and anticorrosive painting application.

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[1] Cepel is a non-profit civil association, a legal entity governed by private law, founded by Eletrobras.

- ✓ Research, development and testing of new technologies of organic coatings.
- ✓ Standardized quality control tests for organic coatings and galvanized steel.

The corrosion laboratory facilities have an advanced equipment infrastructure to perform cutting edge corrosion control research and technological services, including:

- ✓ Potentiostats, Faraday cages, electrochemical cells and electrodes for electrochemical testing.
- ✓ Accelerated corrosion chambers (salt spray, SO_2, UV and condensation) and outdoor natural weathering testing sites.
- ✓ Several equipment for surface preparation and coating application (dry abrasive blasting, air and airless painting sprayers, paint booth, surface profile and dry thickness gauges).
- ✓ Traditional coating testing equipment (abrasion tester, impact resistance, pull-off adhesion, pendulum hardness, glossmeter and viscometers).
- ✓ Conventional laboratorial equipment for chemical reagents weighing and handling (analytical balances and fume hoods).

This paper highlights how this different equipment is used in the day-to-day life of the corrosion laboratory of Cepel for corrosion testing.

3. Research and Testing Purposes

Carbon steel is one of the most used materials for structural purposes in the electric sector, given its good mechanical properties and cost-effectiveness. However, due to its high susceptibility to corrosion, specification of corrosion protection methods for carbon steel is required. In the power industry, the use of galvanized steel and anticorrosive paintings are two of the most employed strategies. The next sections present detailed studies on steel corrosion control methods, testing and experimental research tasks, which the laboratory deals with.

3.1. Surface and Sample Preparation

As corrosion takes place at the interface between a metal and its environment, it is strongly dependent on the material surface conditions. Therefore, the accuracy and reliability of corrosion test results depend on the quality of surface preparation. Furthermore, if the material is to be painted, proper surface preparation is a technical requirement to promote good coating adhesion and anticorrosive performance. It includes a set of activities to clean the surface, remove oxides, paints and other undesired contaminants from it and generate a surface profile [2].

Usually, preparing specimens for any corrosion test involves cutting or shaping the specimen to the desired dimensions and properly labeling the samples before finally carrying out the appropriate surface preparation method. This selection depends on several factors, including the type of material (type of metal), the condition of the surface (degree of rust), the test to be performed (electrochemical tests, accelerated corrosion tests) and if the metal is to be coated or not. For example, different metals, such as steel, aluminum, copper and galvanized steel require different surface preparation methods. In general, the surface preparation method should be selected to achieve the required surface roughness and cleanliness. For corrosion testing involving bare metals, that is, without coating application, the degree of cleanliness is the main concern of surface preparation. If the metal is to be painted, the surface profile is equally important. For electrochemical tests, guaranteeing a regular surface by polishing is crucial due to the high sensitivity of such test techniques. For metallographic analysis, a flat surface is also important for microscopy, requiring proper polishing. The surface should also be evaluated after preparation to ensure that it meets the required quality standards and that it is suited for the intended application. This may involve visual inspection, roughness profile measurement and residual soluble salts quantification on the surface. After the quality control step, the specimen is then ready for exposure to the corrosive environment or for painting application. These practical examples illustrate that physical processing related to sample preparation, selection and evaluation of surface treatment requires solid theoretical background.

The cleaning process uses chemical and mechanical treatments to remove surface contaminants such as oxides, grease, oil and soluble salts. Chemical cleaning uses acids, alkalis or solvents; whereas mechanical cleaning involves the use of mechanical tools such as wire brushes, abrasive pads or sandpaper to physically remove surface contaminants [3]. A combination of treatment is commonly used for cleaning surfaces. For example, metals are usually first degreased with chemical solvents, followed by a mechanical treatment to remove residual oxides. Furthermore, highly adhered oxides not easily removed by some of the mechanical tools may firstly be removed with an acid pickling solution, followed by mechanical means. In the case of anticorrosive paintings, as coating performance is heavily dependent on the quality of surface preparation, the selection of surface treatment is even more important than in other applications.

When it comes to surface preparation prior to coating, two different surface treatments are widely employed in the corrosion laboratory, namely dry abrasive blasting and the use of manual and mechanical tools. Abrasive blasting involves the use of compressed air to propel abrasive particles such as steel grit, glass beads and aluminum oxide onto the metallic surface to remove contaminants and create a surface profile. It is considered one of the best surface preparation methods in the painting industry, as it is possible to achieve a complete removal of residual oxides and generate a proper surface roughness for coating adhesion. However, when it comes to the application of coatings in the electric sector, due to complicated logistics regarding the access to structures and safety restrictions to the use of conductive particles near electric equipment, it is not always possible to employ abrasive blasting, especially in the case of a maintenance coating application [4]. Bearing this in mind, the corrosion laboratory also works with mechanical and hand tools, such as sanding machines and steel brushes, for surface preparation, simulating practical onsite applications in the electric sector field. **Figure 1** and **Figure 2** illustrate different surface preparations and their respective degrees of cleanliness and surface profiles. Cross-section micrographs of coated samples with different surface treatments highlight the better surface profile generated by abrasive blasting. On the other hand, the use of power-tools alone, despite providing a higher degree of cleanliness than manual tools, tends to produce a flat surface, which is far from ideal for coating adhesion.

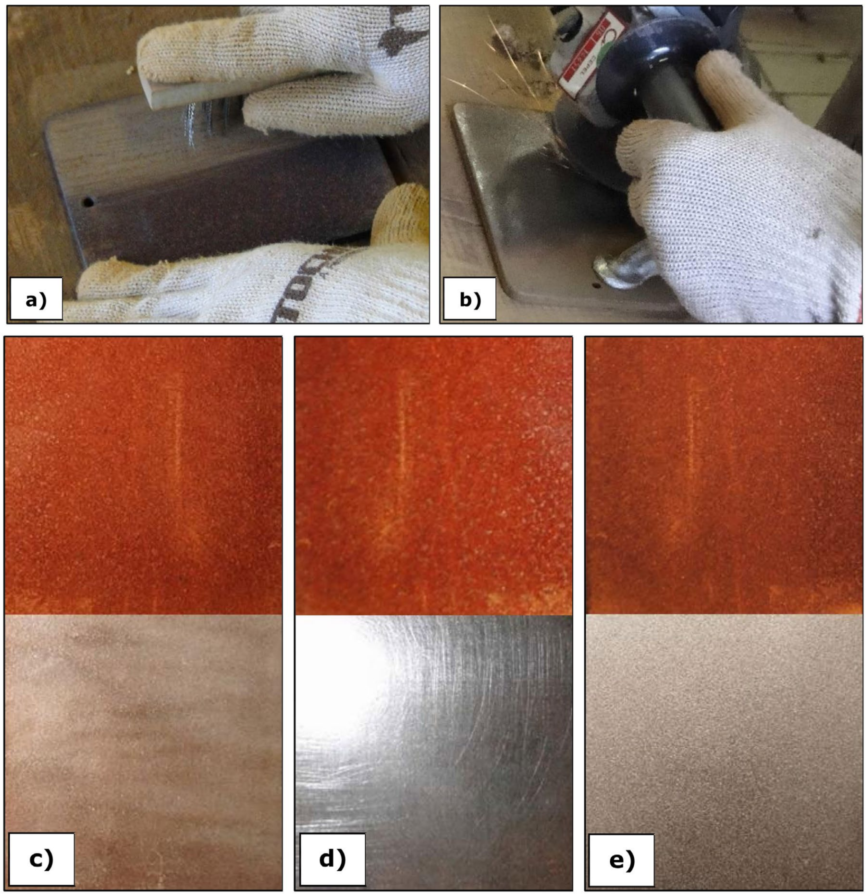

Figure 1. Surface preparation and cleaning of metals: (a) and (c) hand tool cleaning; (b) and (d) power-tool cleaning and e) white metal blasting.

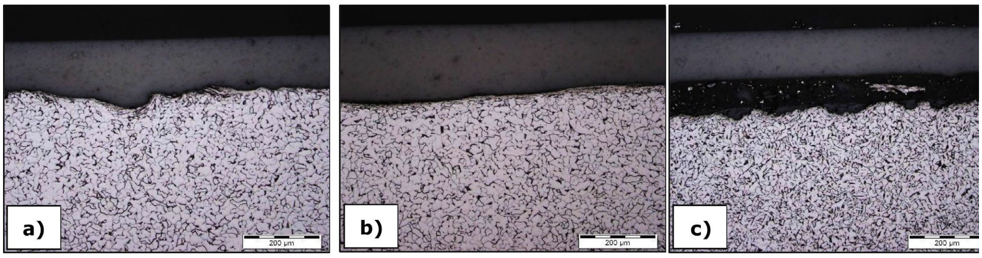

Figure 2. Examples of cross-section optic microscopy images showing the roughness profile of coated surfaces. Metallic substrates were prepared by (a) hand tool cleaning; (b) power-tool cleaning and (c) abrasive blasting.

Considering what was discussed in this section, the following activities summarize the day-to-day life in the corrosion laboratory involving steel surface preparation prior to coating:

1) Cutting and shaping samples to desired dimensions.

2) Degreasing samples with organic solvents to remove oils and greases impurities.

3) Acid pickling to remove the excess of corrosion products or mill scale.

4) Carrying out the method of choice for surface preparation (abrasive blasting or the use of mechanical or manual tools).

5) Measuring the surface profile.

6) Visual inspection for assessing and registering the degree of cleanliness.

7) Storing samples in desiccator filled with silica gel and under vacuum atmosphere to prevent further oxidation.

If an electrochemical test is to be performed, the set of activities listed from 1 through 3 are similar, however, the remaining of routinely conducted tasks for sample preparation can change significantly, as it follows:

4) Soldering a conductive metal wire (usually copper or aluminum) necessary for electrical contact of the electrode in the electrochemical cell.

5) Embedding the sample in epoxy resin to isolate the electrical contact and facilitate sample preparation.

6) Wet polishing embedded sample with sequentially higher sandpaper grits using an automatic polisher.

7) Washing samples with deionized water, acetone and drying with compressed air.

8) Storing samples in desiccator filled with silica gel and under vacuum atmosphere to prevent further oxidation.

These examples highlight how surface and sample preparation depend heavily on the intended application for the material.

3.2. Organic Coatings

Anticorrosive paintings are polymeric coatings designed to protect metallic surfaces from corrosion by providing a barrier that isolates the substrate from the environment and its corrosive agents. The painting system may be comprised of several coats, including a primer, intermediate and top coating, each of them with specific properties and functions [3].

The team of the corrosion laboratory of Cepel, based on over 25 years of research on anticorrosive coatings and related services, worked on a project demanded by its main funding member, Eletrobras, in partnership with Eletrobras technical team, which resulted in the publication of 29 technical documents on anticorrosive painting. Known as Eletrobras Anticorrosive Painting Standards, these publications have been updated continuously by Cepel's corrosion and Eletrobras teams, and used mainly by the Brazilian electric sector to guide proper coating specification, selection, application and quality control to ensure expected long-term painting performance. The set of standards has been tailored to the specific requirements of the Brazilian electric sector, based on Cepel's corrosion research expertise, Eletrobras field application and several international standards [5].

Coatings are selected based on the type and substrate condition, the corrosive environment and the desired service life of the coating system. Besides guiding the proper coating technical specification, Eletrobras Anticorrosive Painting Standards specify requirements to achieve the expected lifetime, such as coating thickness, surface preparation method and laboratorial performance parameters. Brazilian utilities can use the standards for inspection purposes during coating application for quality control, as well as request painting tests for Cepel's corrosion laboratory to check whether the standard requirements are being met. Companies in the electric sector have recognized that the proper use of anticorrosive painting standards is essential for the effectiveness of corrosion protection of their assets.

3.2.1. Coating Application

When it comes to coating application, several steps need to be conducted to guarantee expected coating performance, starting from proper surface preparation and ending with dry film quality control. As surface preparation has already been previously discussed, below is a list of the activities performed by the corrosion laboratory team during coating application to ensure that desired coating properties are met:

1) Checking if the paint gallon presents any damage to the can and if its identification matches the coating to be applied.

2) Mixing the gallon effectively to homogenize its contents.

3) If it is a two-package coating (2 k *coatings*), the components are mixed in amounts that respect the stoichiometric ratio specified by the manufacturer.

4) If necessary, paintings are diluted using the diluent specified in the product's technical data sheet.

5) Waiting for the coating induction time, usually 15 to 20 minutes after mixing the components.

6) Monitoring environmental conditions such as relative humidity, air temperature, surface temperature and dew point, making sure they are proper for coating application.

7) Applying the coating with the proper application method.

8) Controlling the wet coating thickness and guaranteeing it is in range with specifications by the manufacturer.

9) Measuring the coating dry thickness after the dry to handle time is reached.

10) Waiting for the total curing time as specified by the manufacturer.

Based on the anticorrosive standards and the specified procedures, the laboratory provides training programs on the proper selection, application and inspection of anticorrosive coatings. **Figure 3** illustrates some of the activities performed during one of the training programs, including wet and dry thickness measurements, which are important to guarantee the expected coating performance.

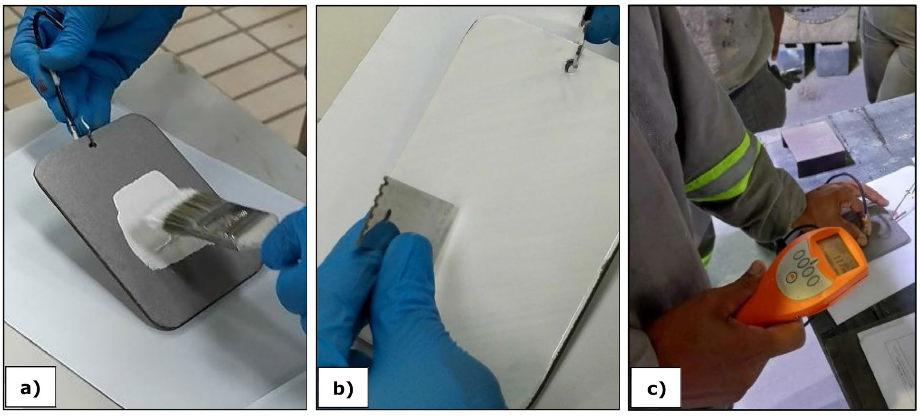

Figure 3. Application of anticorrosive coating by brush (a); wet (b) and dry (c) thickness measurement.

An example was carried out during the training program.

3.2.2. Testing of Commercial Anticorrosive Coatings

Coating quality control can be divided into two approaches: liquid paintings and dry film analysis. The main tests that can be used for liquid paintings are viscosity, pot life, percent of volatile organic compounds (VOC), density and sag test. Adhesion, thickness and drying times are the main parameters that can be analyzed in the dry films. Depending on the application, abrasion and impact resistance, coating flexibility and hardness are other useful tests that can be performed in the dry film. Moreover, the developed set of standards also specifies several test methods and procedures for evaluating coating performance, including accelerated corrosion tests [5]. These tests are routinely conducted by the corrosion laboratory as technological services requested by the companies of the Brazilian electric sector or as part of research projects. **Figure 4** illustrates liquid paintings analysis conducted by the laboratory on commercial paintings as part of a requested technological service. Depicted tests consist of VOC determination and sag test, that is, the maximum wet coat thickness without the risk of sagging when the coating is applied on vertical surfaces. **Figure 5** illustrates the pull-off adhesion test of cured coatings, the most widely used method for evaluating coating adhesion. The test involves attaching a loading fixture to the coating surface, applying a tensile force and measuring the force required to pull the coating off the substrate. The test results can be used to evaluate the strength of the bond between the coating and substrate and to identify any weak adhesion throughout the layers [6]. If a coating has a low adhesion to the substrate, it is more susceptible to premature service failure.

Each test has its own set of specific activities that need to be properly performed, based on international standards and the expertise of the corrosion laboratory team. To illustrate this, a list of tasks related to the day-to-day life in the corrosion laboratory when preparing and executing the pull-off test is shared bellow:

1) Preparing the loading fixture, usually aluminum dollies, by subjecting it to chemical cleaning with organic solvents to remove existing paints and light abra-

sive blasting it to open a surface profile.

2) Selecting at least three areas to attach the dollies, based on coating thickness uniformity.

3) Lightly abrading the areas to be assessed with sandpaper to improve dolly adhesion.

4) Removing particles and dust generated after abrading the coating with appropriate solvents.

5) Preparing the adhesive to glue the dollies, usually a two-component epoxy, in accordance with manufacturer's recommendations.

6) Applying the adhesive to the dollies and attaching them to the coating.

7) Removing the excess adhesive from around the dollies.

8) Waiting for the adhesive curing time.

9) Performing the pull-off test with an adhesion automatic tester.

10) Writing down the pull-off strength and designating the percentage of adhesive and/or cohesive failures within each layer they occur.

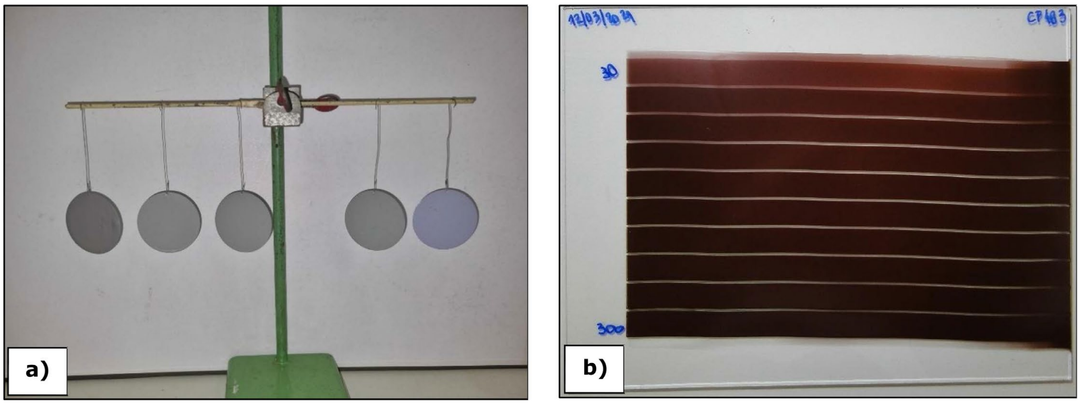

Figure 4. Liquid paintings analysis of commercial coatings requested by the companies of the Brazilian electric sector: (a) VOC determination and (b) sag test.

Figure 5. Evaluation of coating adhesion using the pull-off method.

3.2.3. Development of New Paintings Technologies

The corrosion laboratory also carries out projects regarding the development of new painting technologies, namely smart coatings with self-healing properties. This new generation of paintings has drawn attention as they can provide anticorrosive protection to the substrate even when the painting is damaged. Rather than act only as a barrier between the substrate and the environment, smart coatings are designed to react to the environment and provide active protection, triggering the ability of self-healing upon damage, enhancing coating life and the time for first maintenance [7]. The Cepel's corrosion laboratory has worked on developing self-healing coatings by synthesizing microcapsules containing healing agents. These microcapsules rupture when the coating is damaged, releasing the healing agent that forms a self-healing film at the coating defect. **Figure 6** shows scanning electron microscopy of the microcapsules and how they reduce the corrosion around the scribe made to simulate a damaged coating.

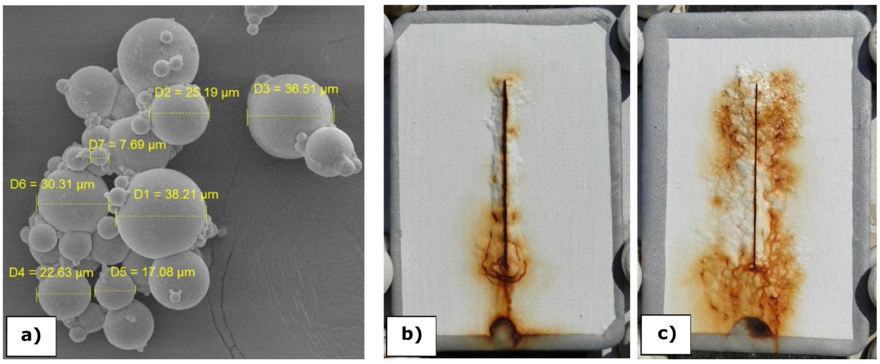

Figure 6. Microcapsules for smart coatings. (a) Scanning electron microscopy image of microcapsules. Corrosion around the scribe in (b) self-healing coating after 2 years of atmospheric exposure; and (c) conventional coating after 2 years of atmospheric exposure.

3.3. Electrochemical Techniques

Corrosion is sustained by charge transfer reactions happening at the interface of the metallic substrate and the corrosive environment. Therefore, electrochemical techniques are used to better understand corrosion processes [8]. Electrochemical tests are usually carried out by means of three-electrode cells connected to a potentiostat. Useful techniques include potential measurements, polarization curves and impedance spectroscopy. These tests can provide valuable information on the corrosion behavior and protective properties of metallic materials, coatings and corrosion inhibitors.

One of the most used electrochemical methods for corrosion evaluation is the polarization technique, which involves the measurement of the current and potential response of an electrode in solution. The higher the current, the faster are the charge transfer reactions, indicating that the metal is corroding at a faster rate. This technique can be used to access the corrosion rate of different metallic materials and to access the performance of corrosion inhibitors. Impedance spectroscopy is another electrochemical technique widely used, especially when it comes to anticorrosive coatings. This method can give insight into the barrier properties of coatings, identifying which painting is more suited for isolating the metal from corrosion.

Figure 7 shows anodic polarization curves of steel in saline solution under different concentrations of a commercial corrosion inhibitor. This was part of a service performed by the corrosion laboratory of Cepel that assessed the use of such inhibitor in hydroblasting surface preparation to prevent the steel from suffering a process known as flash rust. All the curves containing the inhibitor were displaced to lower anodic current densities, in comparison with the saline solution without inhibitor, reducing the corrosion rate of steel. The curves point out an optimum concentration between 1% and 2.5% of inhibitor for maximum corrosion protection of the bare metal. These results highlight that electrochemical tests are powerful tools and play a key role in properly designing anticorrosive methods.

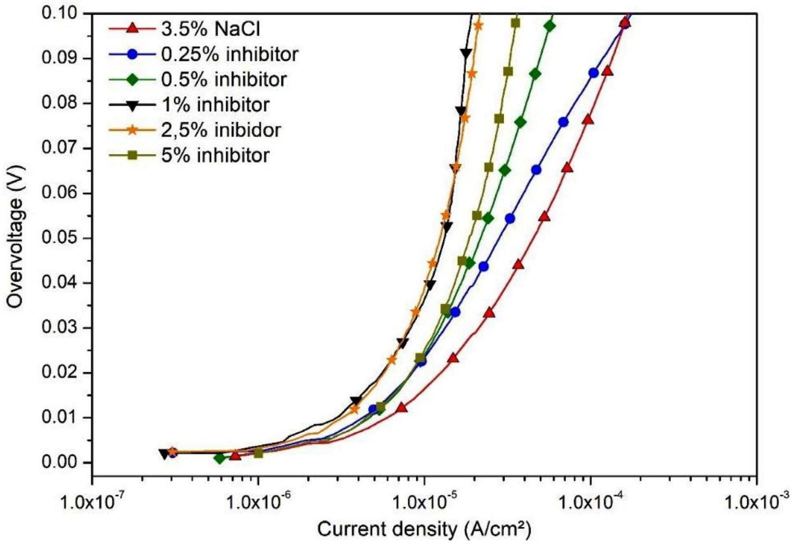

Figure 7. Anodic polarization curves of steel in 3.5% wt. NaCl solution under different concentrations of corrosion inhibitor.

Typical tasks associated with electrochemical testing in the corrosion laboratory are listed below:

1) Selecting the electrodes to be used: working electrode (sample), reference electrode (Ag/AgClsat or saturated calomel electrode) and counter electrode (platinum mesh or graphite rods).

2) Preparing the working electrode according to procedures listed at the end of section 3.1.

3) Measuring the surface area of the working electrode.

4) Preparing the electrolyte solution (usually NaCl 3.5%).

5) Assembling the electrochemical cell, with appropriate positioning of the electrodes to minimize the ohmic drop.

6) Mounting the electrochemical cell inside a Faraday cage to minimize external interferences.

7) Connecting the cell to the potentiostat leads.

8) Configuring the electrochemical procedure in the potentiostat's software.

9) Exporting the results and plotting them with appropriate graphical software.

3.4. Accelerated Corrosion Tests

Depending on the metal and its protective system performance, corrosion processes can be slow and take several years to obtain conclusive results in natural weathering. This is where accelerated corrosion tests come into play, such as salt spray, UV-condensation and acid rain tests, by which extremely aggressive conditions are created, allowing the corrosion process assessment in a matter of days or months.

One of the most used accelerated corrosion tests in the Cepel laboratory is the salt spray (fog) test, which involves the exposure of specimens to a corrosive environment consisting of a saline solution sprayed onto the specimens at a controlled temperature and humidity, simulating marine atmospheres [9]. The combination of a saline medium, saturated humidity and a warm temperature creates a highly corrosive environment. The duration of the salt spray test depends on the intended application of specimens, which can be around 240 hours for bare metals to more than 720 hours (1 month) for anticorrosive coatings. During this time, specimens are periodically inspected and evaluated for signs of corrosion, including rust formation, blistering and corrosion around the scribe.

Results obtained from the salt spray test can be used to determine the corrosion resistance of metals and to assess coating performance. However, it is important to note that there is no linear relationship between the time to rust in salt spray and the lifetime of the material under actual service conditions, since the test cannot replicate the complex environments conditions that specimens are exposed in natural weathering. Therefore, the testing is best suited as a comparative measurement between different materials or protective systems under the same conditions. The UV-condensation test is used to evaluate the weathering resistance of coatings and other materials and involves exposing test specimens to cycles of ultraviolet (UV) radiation and condensation at controlled temperature and humidity [10]. The UV exposure simulates the effects of sunlight, while the condensation cycle simulates the effects of moisture on the specimens. The test is typically performed in UV chambers using a Xenon arc lamp or specific UV radiation lamps (UV-A or UV-B), which emit UV radiation spectrum that closely resembles spectral distributions of sunlight. The UV exposure causes degradation of the polymeric coating, while the condensation allows for water ingress and substrate corrosion. The results obtained from the UV-condensation test can be used to evaluate the durability and weathering resistance of coatings and other materials under outdoor exposure. Gloss retention and degree of chalking are two of the main parameters evaluated during this test.

The Kesternich test, also known as SO_2 chamber, is used to evaluate the resistance of metallic materials and coatings to acid rain. This test involves exposing samples to sulfur dioxide (SO_2) and humidity, which simulates the effects of acid rain by producing sulfuric acid [11]. This test is particularly useful in evaluating the performance of materials and coatings for outdoor applications in industrial or urban environments, where acid rain or SO_2 emissions may be a concern. **Figure 8** illustrates the equipment for the different accelerates corrosion tests previously discussed.

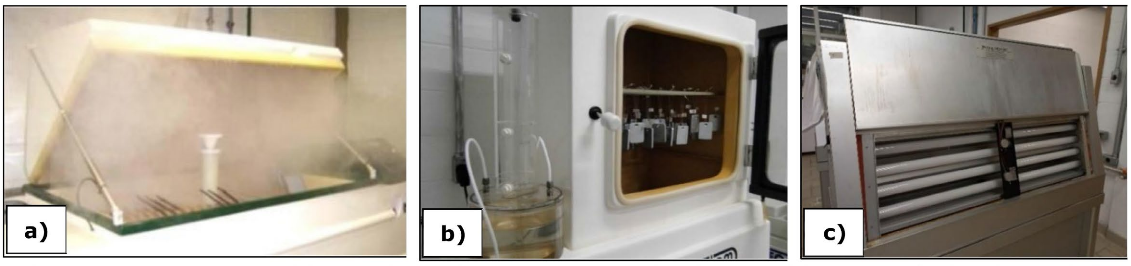

Figure 8. Equipment used for accelerated corrosion tests: (a) salt spray, (b) Kesternich chamber and (c) UV chamber.

Besides each individual accelerated corrosion test, cyclic tests that combine exposure in more than one accelerated chamber are usually performed to better simulate natural conditions. One of the most useful cyclic tests involves the exposure of protective coatings to salt spray test, UV-condensation chamber and a freezer at negative Celsius-scale temperatures [12]. Cyclic tests can be even more aggressive than continuous accelerated tests and usually have a better correlation with natural weathering results.

Each accelerated corrosion test has its own set of parameters to be monitored, in order to control the exposure conditions according to international standards. When it comes to the operation of the salt spray chamber, the following tasks are performed by the corrosion laboratory team:

1) Preparing the salt solution (NaCl 5%) and filling the equipment's reservoir.

2) Guaranteeing the supply of compressed air and deionized water to the saturator tower.

3) Positioning the samples in racks with a 30˚ from the vertical plane.

4) Placing fog collectors throughout the chamber to monitor fog distribution, quantity and composition.

5) Checking if the volume of condensate collected is between 1 and 2 mL per hour.

6) Measuring the sodium chloride concentration of the collected solution with a salinity refractometer and checking if it is around (5 ± 1)% NaCl.

7) Measuring the pH with a pH meter and checking if it is in the range of 6.5 to 7.2.

8) Conducting periodic visual inspections of samples for signs of corrosion and registering such changes.

3.5. Corrosivity of Atmospheres and Determination of Corrosion Rate

Corrosion can occur in a wide range of atmospheric environments, and the atmospheric degree of corrosivity depends on factors such as temperature, humidity and the presence of pollutants or weathering agents. ISO 9223 provides a classification system for atmospheric corrosivity for standard metals, including carbon steel, zinc, aluminum and copper, ranging from C1 (least corrosive) to C5 (most corrosive) and a newly added CX category (extreme corrosivity) for each metal

[13]. Classifying the atmospheric corrosivity can aid on the proper material selection and coating specification during the design of assets, considering the specific conditions they will be exposed to. For example, materials and coatings that are resistant to corrosion in a C1 environment may not be suited to a more corrosive C4 environment.

Two different methods for classifying the corrosivity of atmospheres are covered by ISO 9223, namely a classification in terms of environmental pollution or based on corrosion rate data. The former requires the measurement of the time of wetness and pollution by sulfur dioxide and airborne salinity, estimating the corrosion rate by dose-response functions. The latter is the preferred method, as corrosion rate is directly determined through mass loss results, reducing the level of uncertainty [13]. Mass loss tests involve exposing a standard metal of known weight to a corrosive environment. After exposure for at least one year, the specimen is taken to the laboratory, cleaned and weighed to determine the amount of material lost due to corrosion. The corrosion rate is then calculated by dividing the weight loss by the exposure time and by the surface area of the specimen. **Figure 9** illustrates the determination of the atmospheric degree of corrosivity at the coast of the State of Rio de Janeiro, in Brazil, by mass loss procedures for standard metals. ISO 9226 specifies the procedures for cleaning the samples, based on chemical pickling solutions that effectively remove corrosion products without attacking the underlying substrate [14].

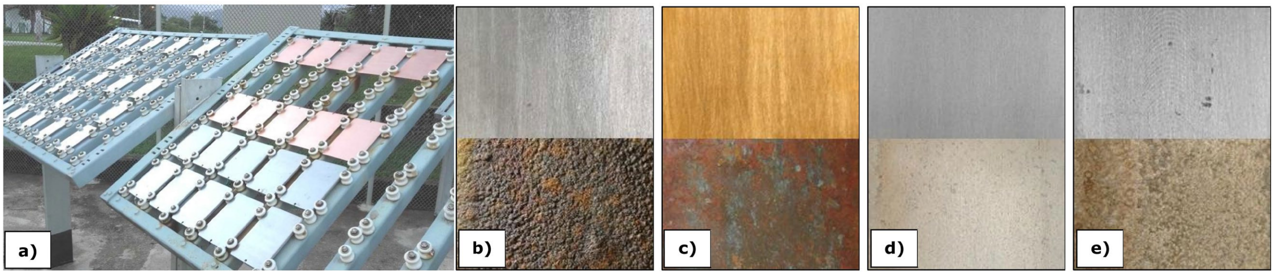

Figure 9. Determination of corrosion rate of standard specimens for the evaluation of corrosivity: (a) exposure site; (b) carbon steel; (c) copper; (d) aluminum and (e) zinc before and after cleaning procedure.

Based on aforementioned international standards, the corrosion laboratory team performs the following tasks when conducting mass loss procedures:

1) Cleaning specimens by chemical means (degreasing and pickling to remove oils and oxides, respectively).

2) Sanding the samples to uniformize the surface of replicates.

3) Measuring the surface area of specimens with a caliper.

4) Determining the initial weight of samples in analytical balances with readability of 0.1 mg.

5) Properly labeling the samples.

6) Exposing specimens to corrosive media.

7) Inspecting and registering visual aspects of samples by the end of exposure.

8) Determining specimens' weights after exposure.

9) Subjecting samples to sequential steps of cleaning by pickling solutions.

10) Monitoring changes in weight after each cleaning step.

11) Plotting a graphic of mass loss vs number of cleaning cycles.

12) Determining the end of cleaning procedure when a nearly horizontal line in the plot of mass loss is achieved.

13) Determining final weight of metal by graphical means.

14) Calculating corrosion rate through weight loss, time of exposure and surface area of specimens.

3.6. Soil Corrosion

Soil can be a particularly corrosive environment due to the presence of moisture, dissolved salts and microorganisms. Furthermore, at the interface between the soil and the atmosphere, the corrosion process is intensified for structures that are partially buried, due to a phenomenon called differential aeration corrosion [1]. In the electric sector, the transmission tower foundations are susceptible to soil corrosion. In order to effectively prevent and mitigate soil corrosion, it is important to understand the corrosivity of the soil environment.

One key factor in determining the soil corrosivity is its resistivity, which is a measure of its ability to oppose the flow of ionic current. Metals buried in soils with low resistivities are more susceptible to corrosion. Soil resistivity is usually determined by the Wenner method. This procedure involves placing four electrodes in the soil in a straight line and connecting them to an earth tester, which measures the soil resistance. Knowing the electrode spacing, soil resistivity can be calculated from resistance measurements [15]. **Figure 10** illustrates the resistivity determination at a field inspection conducted by the corrosion laboratory in a wind park in the northeast of Brazil.

Figure 10. Wenner method for soil resistivity determination in a Brazilian wind park.

The soil characterization also includes tests to evaluate the pH, soluble salts, moisture content and organic matter content of soil samples, requiring the support from the chemical laboratory to carry out these analyses. By analyzing these properties, it is possible to gain a better understanding of the corrosivity of a soil and to develop effective corrosion prevention and mitigation strategies. Figure 11 shows the difference between anticorrosive performance of different metals in soil, highlighting that soil corrosion cannot be taken for granted.

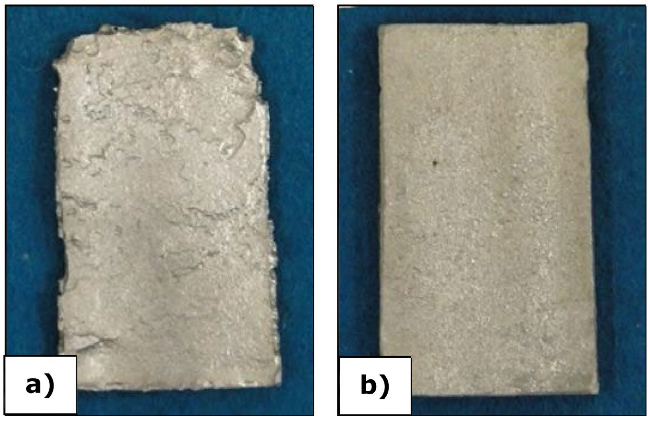

Figure 11. Corroded specimens after chemical cleaning exposed for four years to a soil contaminated with sodium chloride: (a) carbon steel and (b) galvanized steel.

The following activities are related to the day-to-day life of the corrosion laboratory when monitoring important parameters for soil corrosivity:

1) Measuring soil resistivity by the Wenner method, using an earth tester connected to four electrodes placed in the soil.

2) Collecting several soil samples for chemical characterization.

3) Measuring soil moisture content by determining the mass loss before and after drying the samples in an oven.

4) Preparing soil aqueous extracts.

5) Measuring pH and conductivity of the extracts.

6) Requesting chloride and sulfate analysis in the soil extract to the chemical laboratory.

3.7. Quality Control of Galvanized Steel

Galvanized steel is one of the most used materials in the electric sector, given its use in the transmission towers and in other equipment in energy generation plants and electric substations. The zinc coating of galvanized steel acts as a sacrificial layer for the underlying substrate, protecting the steel from corrosion by the principles of cathodic protection. Furthermore, corrosion rate of zinc is slow in most environments, providing galvanized steel structures with decades of lifetime, depending on the coating thickness and the corrosivity of the atmosphere [16]. Cepel's corrosion laboratory often performs tests for quality control of galvanized steel, including the determination of coating mass, thickness and uniformity.

Coating mass and thickness give important information regarding the amount of zinc available to protect the steel against corrosion, being useful for designing the service lifetime of galvanized steel structures. Coating thickness can be measured by thickness gauges operating by electromagnetic induction principle [17].

Coating mass can be determined by gravimetric tests involving the dissolution of the zinc layer by acid immersion of a sample with known weight [18]. International standards specify the minimum amount of zinc coating mass, depending on the sample geometry [19]. Preece test, also known as uniformity test, is another method for evaluating the quality of galvanized steel. In this test, the galvanized sample is subjected to immersion cycles in a saturated copper sulfate solution. If the coating has a defect or presents areas with considerably lower thickness, such regions will be susceptible to the premature deposition of copper. To be considered uniform, the coating must withstand a minimum number of immersions without the formation of an adherent copper deposit [20]. **Figure 12** shows the different tests discussed and below is a list of the main tasks in the corrosion laboratory regarding the testing of galvanized steel:

1) Measuring the average coating thickness of each sample using the magnetic gauge.

2) Selecting specimens that represent well the population of samples to carry out quality control tests that are destructive (coating mass and uniformity).

3) For the coating mass test, after determining the initial weight and area of the sample, the galvanized steel is immersed in a specific hydrochloric acid solution containing corrosion inhibitors for steel to remove the zinc coating without attacking the substrate. Immersion is stopped when the intense evolution of hydrogen has ceased, indicating that all zinc has been removed. The weight difference after zinc removal is determined to calculate the coating mass.

4) For the Preece test, cycles of immersion of the galvanized steel in a saturated copper sulfate solution are conducted, each one followed by brushing the sample with a fiber bristle brush, to remove any non-adherent copper deposits. Cycles are repeated until an adherent deposit is formed.

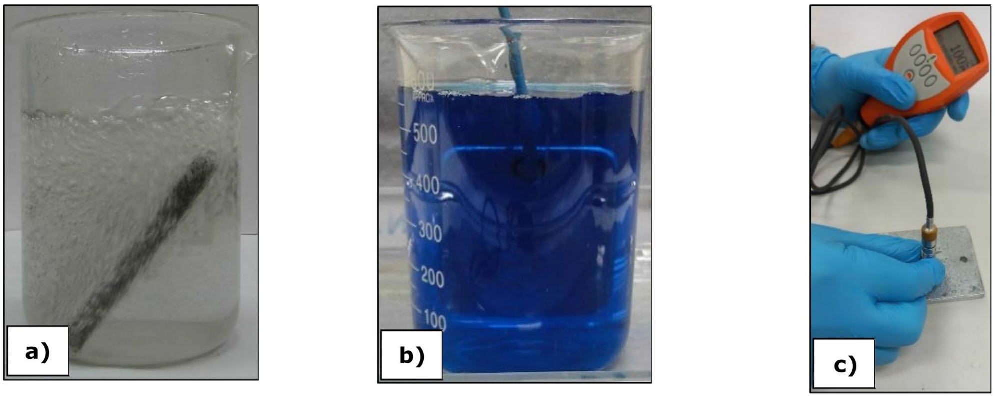

Figure 12. Quality control tests for galvanized steel: (a) coating mass; (b) coating uniformity and (c) coating thickness.

The Cepel's Corrosion Laboratory also conducts research projects on the corrosion performance of galvanized steel, assisting on the specification of this material for the companies of the Brazilian electric sector. One of these projects involved the research on the anticorrosive properties of hot-dip galvanized weathering steel. Weathering steel and galvanized steel have their own individual application. Weathering steel contains alloying elements that favor the formation of a protective rust layer when this steel is exposed to urban atmospheres with pollutants. The approach to combine both methods of protection by galvanizing the weathering steel is a recent technology in Brazil, used namely in compact towers of transmission lines. The anticorrosive properties of weathering steel after the zinc layer is corroded were unknown in the state of the art, which was the fundamental drive for this research. Hot-dip galvanized weathering steel was pickled in a controlled way to remove only the zinc layer, simulating a corrosion process of the metallic coating in real conditions. It was investigated if the anticorrosive properties of the weathering steel were preserved in different aggressive media, compared to the weathering steel that was not galvanized.

Figure 13 illustrates the CEBELCOR test setup and the corrosion rate of weathering steel (WS), galvanized weathering steel after zinc removal (WSRZ), zinc (Zn) and carbon steel (CS) after different times of exposure to this test. The CEBELCOR test consists of simulating the action of natural agents (wet and dry cycles, atmospheric pollutants) in a cyclic manner to promote the formation of the protective rust in weathering steel. Samples are fixed in a rotating axis, being immersed in a sodium bisulfite solution during the wet cycle and submitted to the heat of an incandescent lamp at 40˚C during the dry cycle. This test is regarded as the most suitable accelerated test to simulate natural conditions for protective rust layer formation in the weathering steel [16].

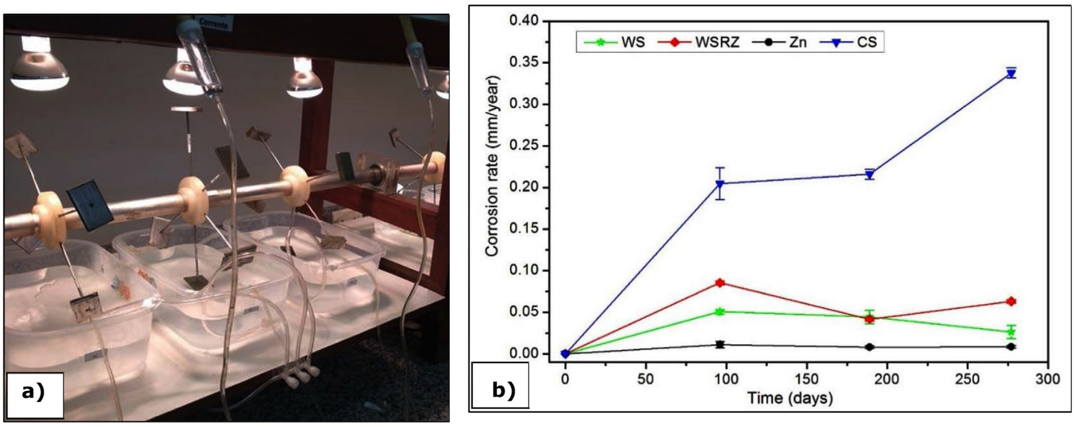

Figure 13. Research project on the anticorrosive properties of galvanized weathering steel: a) CEBELCOR test setup and b) corrosion rate of weathering steel (WS), galvanized weathering steel after zinc removal (WSRZ), zinc (Zn) and carbon steel (CS) after different times of exposure to this test.

The results point out much lower corrosion rates for WS and WSRZ samples than for CS. This suggests that the protective rust layer was properly formed during the test, isolating the steel from the aggressive media, resulting in lower corrosion rates for WS and WSRZ. Therefore, after the zinc layer is corroded in galvanized weathering steel, it is expected that the weathering steel will keep its original anticorrosive properties. It was concluded that the approach to galvanize weathering steel is valid in terms of the additional protection provided to the substrate after the consumption of the zinc layer, given that atmospheric exposure presents proper conditions for the development of a protective rust layer in weathering steel.

4. Limitations

This paper aims to provide a broad overview of the daily activities and practical challenges faced by the corrosion laboratory at Cepel. While technical data such as polarization curves and advanced coating interface analyses are valuable, they fall outside the scope of this article. The focus here is on operational routines and general practices rather than detailed electrochemical or microscopic evaluations. Future publications may address these aspects in greater depth.

5. Conclusion

Corrosion protection is challenging due to its spontaneous nature and its specific characteristic for each metal-environment interaction. There is no universal solution to prevent corrosion, therefore a critical assessment of each individual case is required. With interdisciplinary expertise in materials science, chemistry and electrochemistry, along with an advanced laboratory infrastructure, understanding and controlling corrosion processes becomes possible. This sharing of Cepel's corrosion laboratory experience also aims to contribute to showing the importance of technical-scientific knowledge and practical know-how in the solution or remediation of engineering problems related to corrosion.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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